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SINGLE ION COUNTING IN HIGH-RESOLUTION MASS SPECTROMETRY.(U)

APR 77 F M WACHI, D E GILMARTIN

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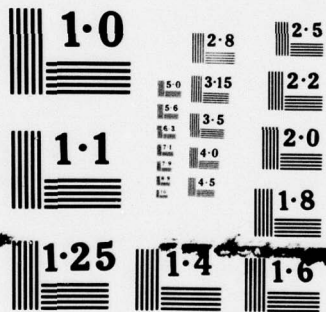
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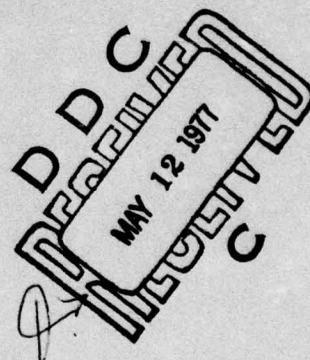
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Single Ion Counting in High-Resolution Mass Spectrometry

Materials Sciences Laboratory
The Ivan A. Getting Laboratories
The Aerospace Corporation
El Segundo, Calif. 90245

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Interim Report



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Project Officer

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PREFACE

The authors wish to thank A. Y. Lu for helpful suggestions and technical assistance.

SINGLE ION COUNTING IN HIGH-RESOLUTION MASS SPECTROMETRY

Recently, mass spectrometric analysis of trace quantities of residual gases in traveling-wave tubes necessitated the development of an ion-counting detector system for the high-resolution mass spectrometer. The spectrometer is a double-focusing instrument of the Mattauch-Herzog geometry (Consolidated Electrodynamics Corp., Model 21-110B) equipped with an ion-detection system consisting of a photoplate, Faraday cup, and electron multiplier. High-resolution mass spectrometry was required to differentiate hydrocarbon fragments from other residual gases such as nitrogen, oxygen, carbon monoxide, argon, carbon dioxide, hydrogen cyanide, nitrous oxide, and nitrogen dioxide. The mass resolution of an ultrasensitive, quadrupole-type residual gas analyzer equipped with an ion-counting detector¹ is inadequate for this application. Furthermore, the need for the quantitative determination of the partial pressures of the residual gases imposed a severe limitation on the use of the direct-current technique of measuring ion currents with the existing electron multiplier because of mass discrimination effects exhibited by the conversion dynode²⁻⁶ and the poor precision and accuracy in measuring the very small ion currents that result from background noise interferences and drift in multiplier gain. A modification that permits the utilization of an air-stable electron multiplier that can be used either as an ion-counting or as an ion-current measuring

device without jeopardizing the photoplate ion-detection capability of the high-resolution mass spectrometer is described in this report.

The original electron multiplier supplied with the mass spectrometer is a 16-stage Allen type with beryllium-copper dynodes. This electron multiplier is inadequate for use in ion-counting techniques because of its inherent high noise level⁷ and its poor frequency response, which precludes the acquisition of data at sampling rates greater than 10 kHz that are required for computer interfacing under high-resolution operation. These limitations have been circumvented by replacing the existing electrical detection system, consisting of the Faraday cup and the Allen-type electron multiplier, with a Channeltron[®]⁸ electron multiplier (Galileo Electro-Optics Corp., Model 4730) assembly⁹ shown in Fig. 1. This detector assembly is attached to the mass spectrometer with a specially designed adapter¹⁰ that aligns the aperture of the Channeltron electron multiplier (CEM) with the collector slit and yet does not jeopardize the photoplate ion-detection capability of the spectrometer. A photograph of the modified combination ion-detection system is shown in Fig. 2.

Although it is not shown in Fig. 1, a 0.002- μ F, 6-kV capacitor is attached between the negative high-voltage biased end of the electron multiplier and instrument ground to remove high-frequency voltage spikes from the power supply. The CEM is protected from stray magnetic fields by the soft iron shield; its potential is also maintained at instrument ground. Electrical power to the CEM is provided by a solid-state, high-voltage power

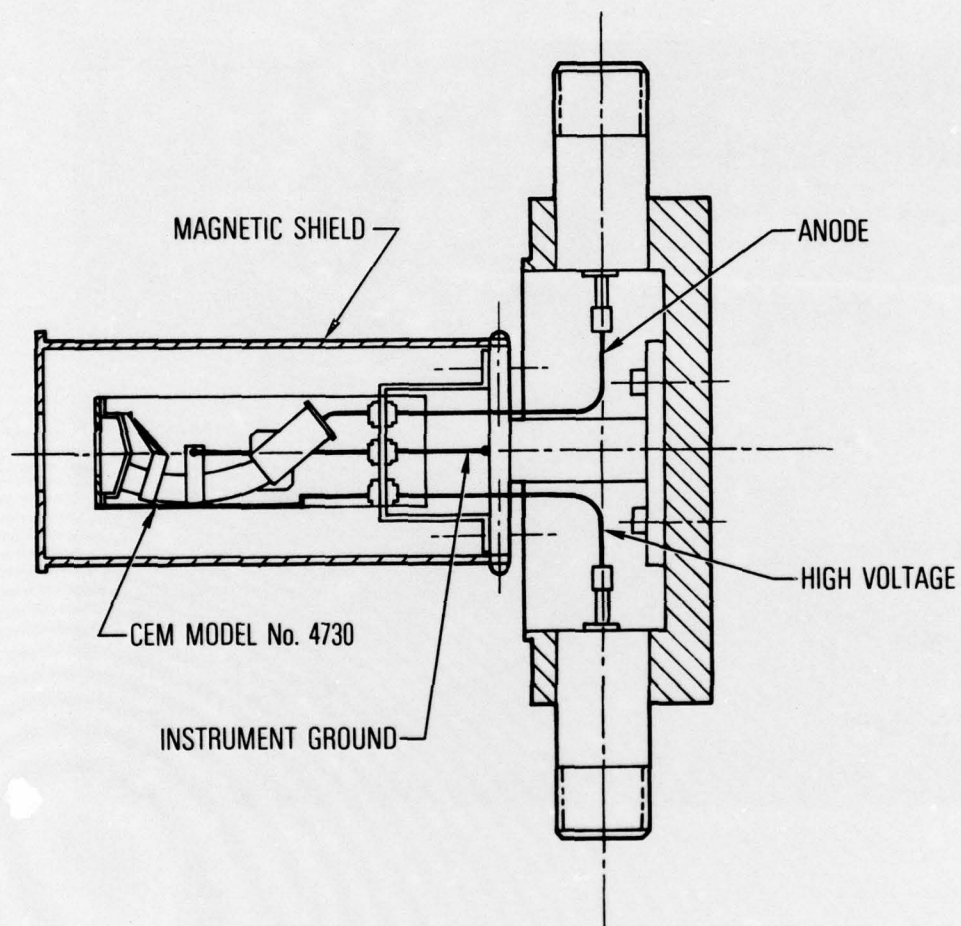


Fig. 1. Schematic of Channeltron Electron Multiplier Assembly

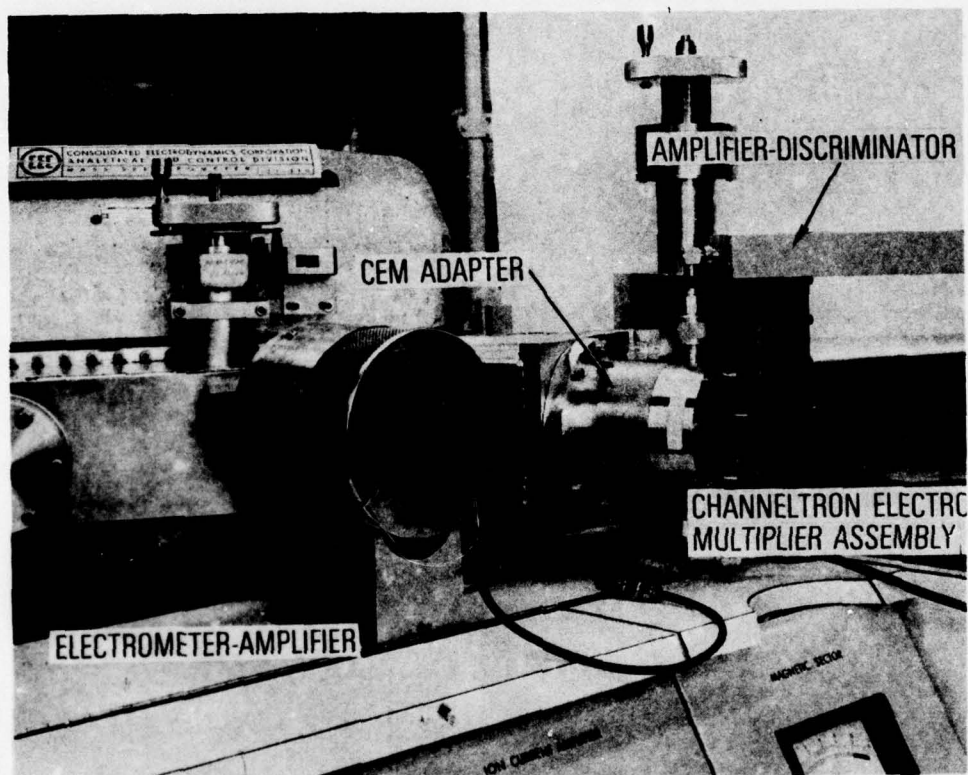


Fig. 2. Modified Combination Ion-Detection System

supply (SSR Instruments Co. , Model 1106-10). The anode of the CEM is connected to the pulse-counting electronic system, consisting of SSR Instruments Model 1120-01 amplifier-discriminator (10-nsec pulse pair resolution) and Model 1110 digital synchronous computer. The digital data are recorded either onto a magnetic tape or a digital recorder for further processing. The performance characteristic of this ion-counting detector system is illustrated in Fig. 3. The multiplier dark count plus the electronic system noise at applied voltages of 2 kV and 3 kV are 0.005 counts/sec and 0.03 counts/sec, respectively. The CEM has exhibited minimal degradation in overall multiplier gain even after exposure to air for over a month during 14 months of operation. The gain measured at an applied voltage of 2.5 kV decreased from 8×10^7 to 4.5×10^7 .

Ion counting not only permits the measurement of very small ion currents with greater precision and accuracy but is also essentially free of effects resulting from mass discrimination and drift due to changes in multiplier gain. Another important feature of this modification is that it permits the rapid conversion from pulse counting to analog mode of ion detection. The conversion consists of removing the amplifier-discriminator and hooking up the electrometer-amplifier whose output may be fed directly into other analog-to-digital converters, i. e. , the original system can be restored in minutes.

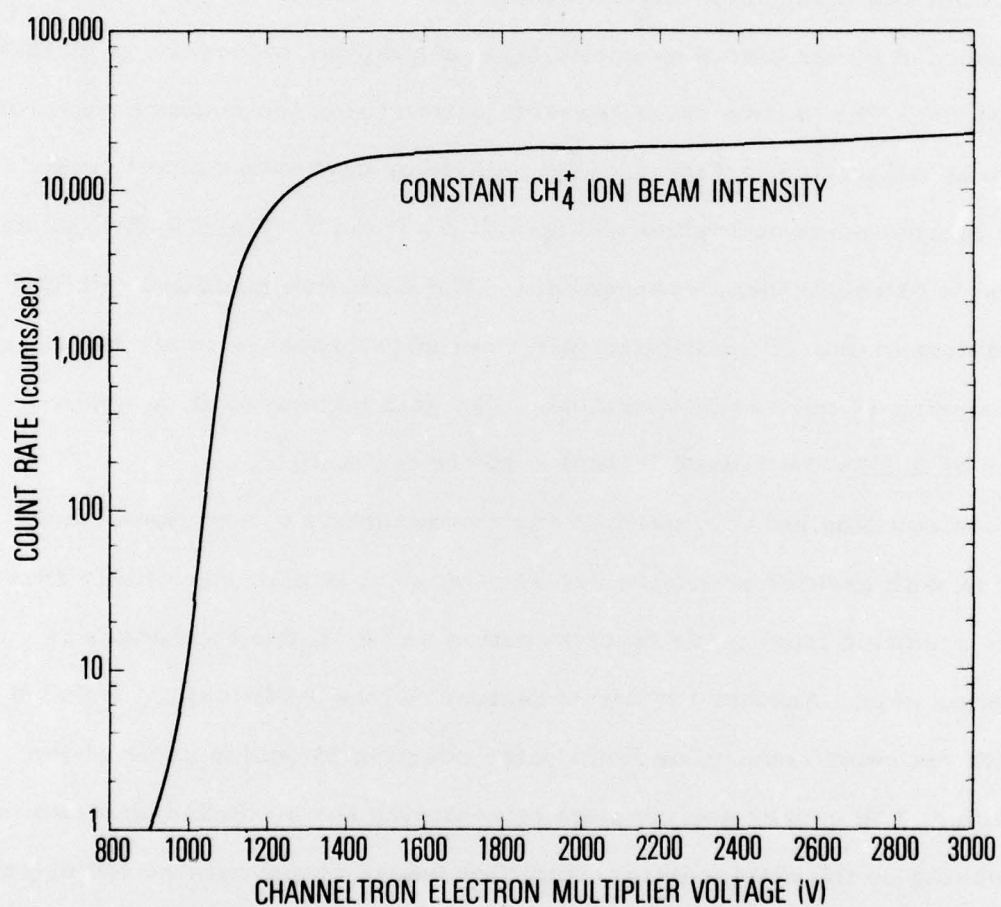


Fig. 3. Count Rate As a Function of Electron Multiplier Voltage

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7. C. A. Stearns and F. J. Kohl, High-Temperature Mass Spectrometry-Vaporization of Group IVB Metal Carbides, NASA TN D-7613, Lewis Research Center, Cleveland, Ohio (April 1974).
8. Continuous-dynode, electrostatic-focusing electron multiplier produced by Galileo Electro-Optics Corp.
9. This entire assembly (Part No. 294790) is manufactured by DuPont Instruments for their Series 21-490 mass spectrometers.
10. Engineering drawings of the adapter (Appendix) may be obtained from one of the authors.

APPENDIX

Assembly instructions for the Channeltron[®] electron multiplier adapter for the mass spectrometer and rework instructions for the model 4730 CEM are presented.

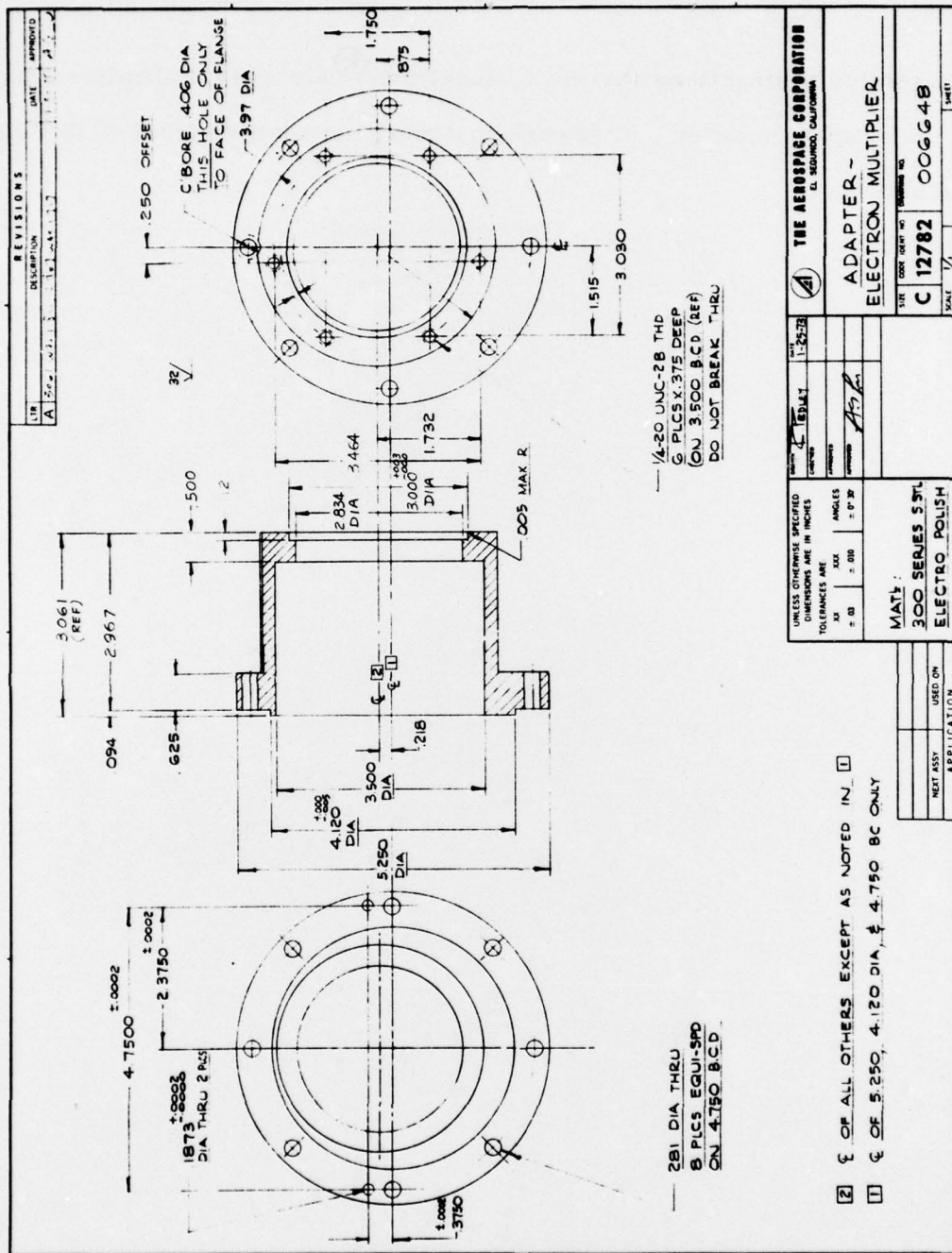


Fig. A-1. CEM Adapter for Mass Spectrometer

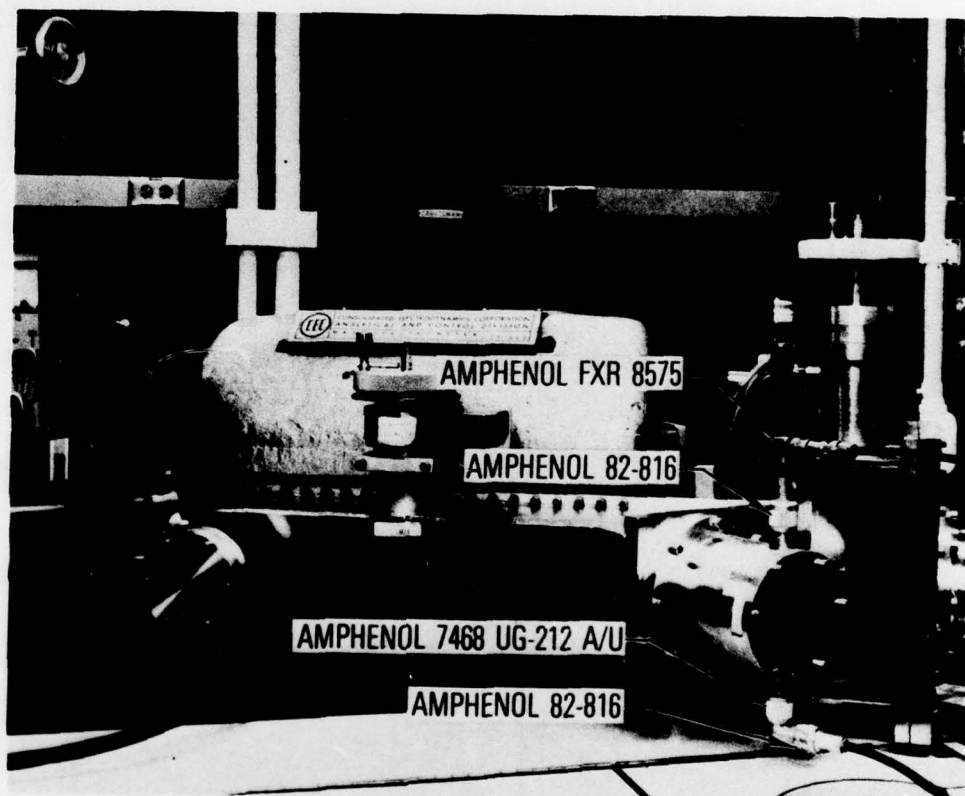


Fig. A-2. Electrical Connectors of CEM Assembly

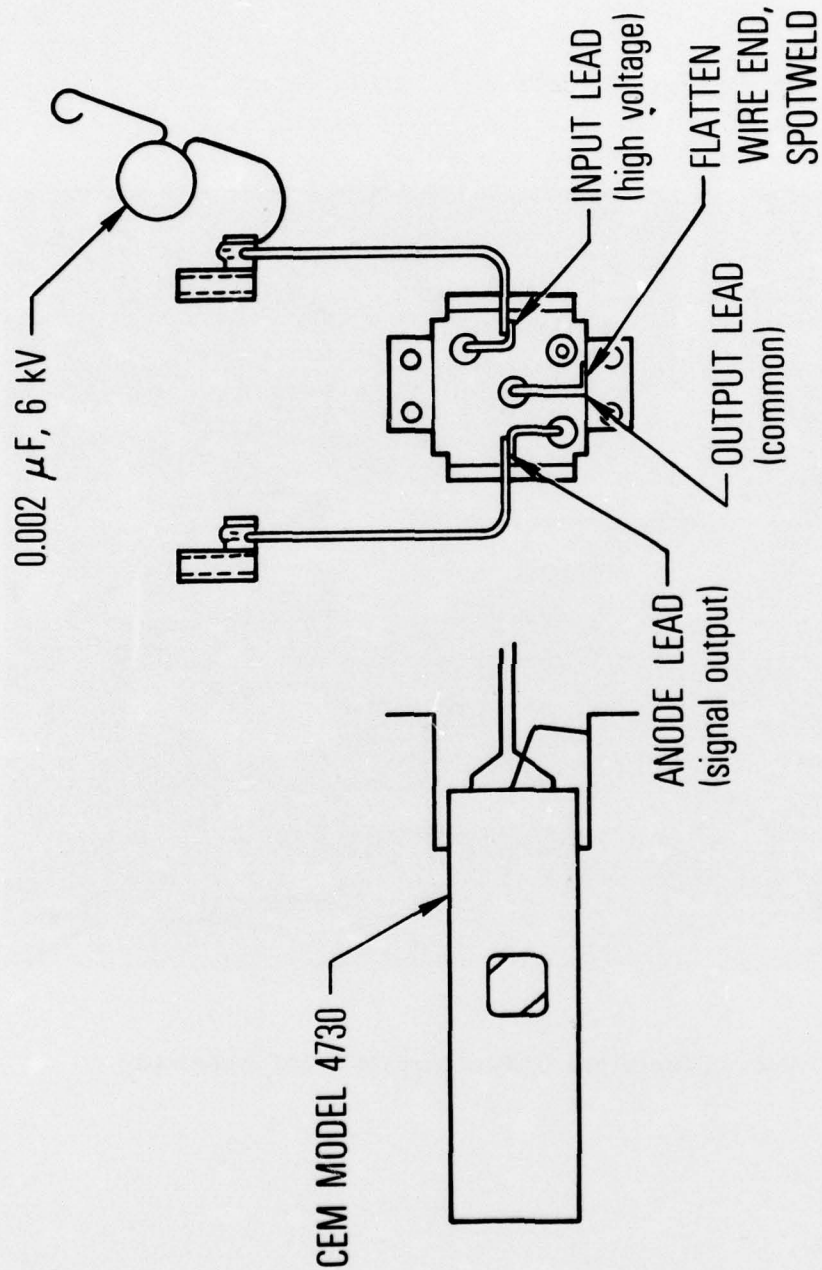


Fig. A-3. Schematic of Channeltron Rework

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